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Microstructure of 1469 aluminum alloy after severe plastic deformation and ion irradiation

N V Gushchina¹, V V Ovchinnikov^{1,2}, F F Makhin'ko¹, L I Kaigorodova³
and D Yu Rasposienko³

¹ Institute of Electrophysics, Ural Branch of Russian Academy of Sciences,
Amundsena Street 106, Yekaterinburg, 620016, Russia

² Ural Federal Technical University named after the First President of Russia B.E.
Yeltsin, Mira Street 19, Yekaterinburg, 620002, Russia

³ Mikheev Institute of Metal Physics, Ural Branch of Russian Academy of Sciences,
St. S. Kovalevskoy 18, Yekaterinburg, 620990, Russia

E-mail: guscha@rambler.ru, gushchina@iep.uran.ru

Abstract. The effect of accelerated Ar⁺ ion beams with an energy of 10 keV on the microstructure and phase composition in 1469 alloy (Al–Cu–Li–Ag–Mg–Zr–Sc) initially subjected to severe plastic deformation (SPD) has been investigated by transmission electron microscopy. Irradiation has been found to result in the formation of a predominantly recrystallized nanocrystalline or fully recrystallized submicrocrystalline structure in the alloy, depending on the ion fluence. The irradiation liquidates the banded structure found in the alloy after SPD. Therefore, the structural elements (nanofragments, nanograins, submicrocrystals) are distributed uniformly in the volume of the irradiated sample. In addition, irradiation increases the volume fraction and the size bimodality of heterogeneously-generated T₂-phase particles. The mechanism of nucleation and growth of excess phases has been proposed. The structure changes at a higher rate than it does during a long low-temperature annealing process, and structural changes are observed at a distance of ~200 μm from the surface, which considerably exceeds the projected ion range (~10 nm).

1. Introduction

The work is aimed at studying the microstructure of the 1469 alloy (Al–3.4Cu–1.2Li–0.46Ag–0.66Mg–0.1Zr–0.08Sc) subjected to severe plastic deformation (SPD) and irradiation with argon ions with an energy of 10 keV.

To study the regularities of ion bombardment on severely deformed materials in order to control the formation of nanocrystalline structure and supersaturated solid solution decomposition is of interest from both fundamental and practical points of view. Despite the available data showing that ion-beam treatment can be used to modify the structure and properties of materials [1, 2], some processes that occur during the exposure to accelerated ion beams are still poorly understood. These processes are largely dependent on the reaction of specific media exposed to irradiation.

The 1469 alloy of the Al–Cu–Li system, one of the most new promising high-strength aluminum-lithium alloys with relatively high copper content and containing Zr, Sc, Mg and Ag additives was chosen as the object of investigation in this work. The alloy was developed at Federal State Unitary Enterprise "VIAM". The alloy has a low density (2.67 g/cm³), high elastic modulus, while successfully



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combining strength and plastic properties, and is characterized by high corrosion resistance and weldability. It is successfully used in industry to manufacture pressed and rolled semifinished products and is recommended for the manufacture of aircraft fuselage elements (wing spars, beams, etc.), long operating under compression at 150°C under all weather conditions.

Severe plastic deformation is an effective way to improve the complex of the properties of Al–Li alloys. The studies carried out before [1, 2] provide grounds to believe that the effects of ion beams on these alloys in combination with SPD can be studied in order to create fundamentally new methods of their processing aimed at providing a unique structure and level of properties that cannot be achieved by using exclusively thermomechanical effects.

2. Experimental

The samples made of 1469 alloy were 2 mm thick. They were deformed at room temperature and a pressure of 4 GPa in Bridgman anvils to 5 revolutions (angle of the anvil rotation $\varphi = 10\pi$ rad). The thickness of the samples after deformation was ~ 400 μm .

The deformed samples were irradiated with continuous Ar^+ ion beams using an ILM-1 ion beam implanter equipped with a PULSAR-1M ion source based on a low-pressure glow discharge with a hollow cold cathode [3]. The Ar^+ ion energy $E = 10$ keV, ion current density $j = 100$ $\mu\text{A}/\text{cm}^2$, and fluence $F = 6.25 \cdot 10^{15}$ and $2 \cdot 10^{16}$ cm^{-2} (irradiation time of 10 and 32 s, respectively) were used. A line-focus beam 2×10 cm^2 in cross section, under which the samples were moved at a speed of 1 cm/s, was cut out from the ion beam of circular cross section using a collimator. This was done to prevent significant integral heating of the targets during irradiation. The temperature of the samples, controlled by thin chromel–alumel thermocouples during irradiation, did not exceed 180°C.

The structure and the phase composition in thin alloy foils were examined using a Tecnai 30G³⁰ Twin electron microscope at the Electron Microscopy Center of Collaborative Access of the Institute of Metal Physics, UB RAS. The foils were made from the samples by thinning them electrolytically from both sides. Thus, the initial and irradiated structures were examined at the center of the samples from a distance of 200 μm from both surfaces.

3. Details of exposure, measurements and results

Electron-microscopic examination of the initial samples has shown that the 1469 alloy after deformation exhibits a partly recrystallized nanocrystalline structure with an average grain diameter of ~ 50 nm (figure 1 *a–c*). This indicates dynamic recrystallization that takes place during SPD. The transition of the deformed alloy into a more equilibrium and partly recrystallized state is accompanied by a decrease in the length of "broken" dipole boundaries. The formation of these boundaries, according to [4, 5], is a relaxation mechanism of the energy stored during severe plastic deformation. Recrystallization decreases their density and length. The nanograins have nonequilibrium convex–concave boundaries. The deformation-induced effects of diffraction contrast in the form of loops or arcs ~ 50 nm in diameter are observed near such boundaries (figure 1 *c*). Analysis of the nature of this contrast indicates the presence of elastically deformed lattice regions [5].

Deformation bands are detected in some parts of the sample, which causes anisotropic distribution of structural elements (nanograins and nanofragments) along crystallographic directions corresponding to the directions of the bands (figure 1 *a, c*).

The superfine T_2 -phase particles with a diameter not exceeding 5–10 nm precipitate heterogeneous at the boundaries of nanoparticles, nanograins, and "fragments" of dipole boundaries during SPD (or subsequent natural aging) (figure 1 *d*).

Electron-microscopic examination of the samples after Ar^+ ion irradiation at a fluence of $6.25 \cdot 10^{15}$ cm^{-2} has indicated a mainly recrystallized structure. The structure consists of nanograins 70–100 nm in diameter and submicrocrystals 0.15–0.2 μm in diameter, which are uniformly distributed (figure 2 *a*). Nanograins and submicrocrystals have an equiaxed shape and equiaxed straight boundaries. The regions with the structure recrystallized are nearly free from dislocations. A small volume of the irradiated sample is occupied by a nonrecrystallized structure consisting of

nanograins up to 100 nm in diameter and nanofragments up to 50 nm in diameter (figure 2 *b*). Nanograins have straight boundaries and nanofragments have convex–concave ones. Incomplete recrystallization in these regions is proven by the presence of dislocation clusters and dipole boundaries (figure 2 *b*).

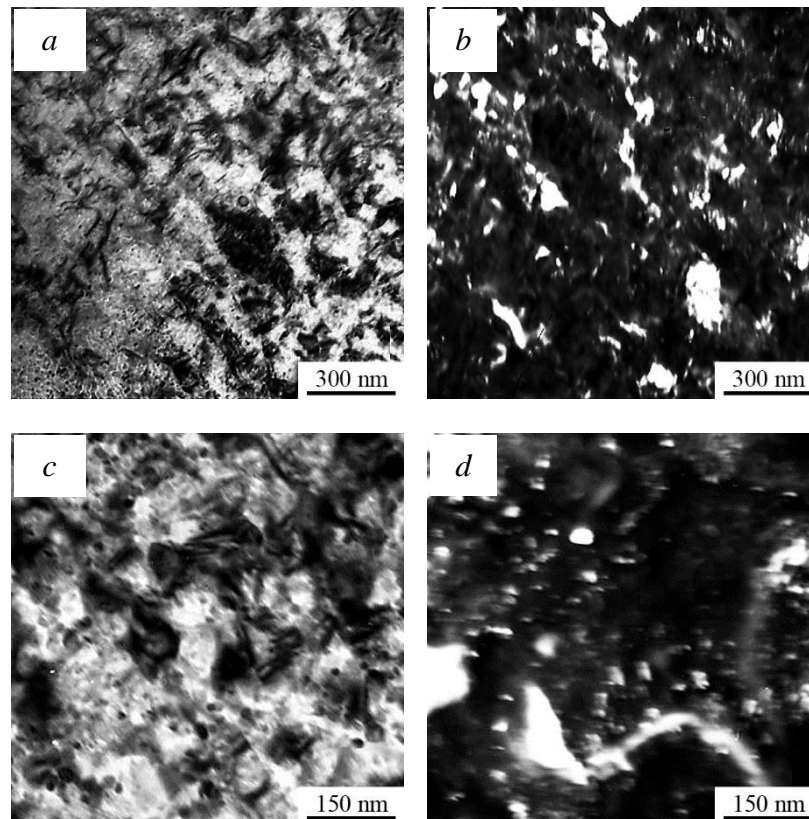


Figure 1. Microstructure of the 1469 alloy after SPD: (*a*) (*c*) bright-field image and (*b*), (*d*) dark-field images: (*b*) in reflection $(200)_{Al}$ and (*d*) in reflections $(111)_{Al}$ and $(530)_{T_2}$.

In summary, Ar^+ ion irradiation at a fluence of $6.25 \cdot 10^{15} \text{ cm}^{-2}$ causes microstructure transformation, but does not eliminate the structural heterogeneity of a severely deformed alloy. This means that regions with recrystallized nanocrystalline and nonrecrystallized nanofragmented structure coexist together [1].

Heterogeneously nucleated T_2 -phase particles are observed at nano and submicrocrystal boundaries after both SPD and irradiation. The particle diameter at submicrocrystal boundaries is 15–20 nm (figure 2 *c*), and 5–10 nm at nanograin boundaries (figure 2 *d*). The kinetics of the formation of nano- and submicrocrystals seems to be responsible for this size distribution of grain boundary precipitates. The fact that the density of coarse precipitates is several times lower than that of fine ones is noticeable.

The size bimodality of T_2 -phase precipitates gives an idea of two ways of their formation. The coarse particles are formed by irradiation due to coagulation of particles with a diameter larger than the critical one under these thermodynamic conditions. The fine T_2 precipitates nucleate during subsequent natural aging of the irradiated sample. The decomposition results in the oversaturation of the solid solution with Cu and Li alloying elements due to dissolution of the phase particles with a diameter smaller than the critical one during irradiation.

An increase in the fluence to $2 \cdot 10^{16} \text{ cm}^{-2}$ facilitates the transition from partially to completely recrystallized homogeneous submicrocrystalline structure (figure 3 *a*). The equilibrium shape, straight lines, and equilibrium triple junctions of submicrocrystals, as well as the absence of dislocation pile-up

in the images of the alloy irradiated indicate its recrystallized state (figure 3 *a*, *b*). The average submicrograin diameter was $\sim 0.3 \mu\text{m}$.

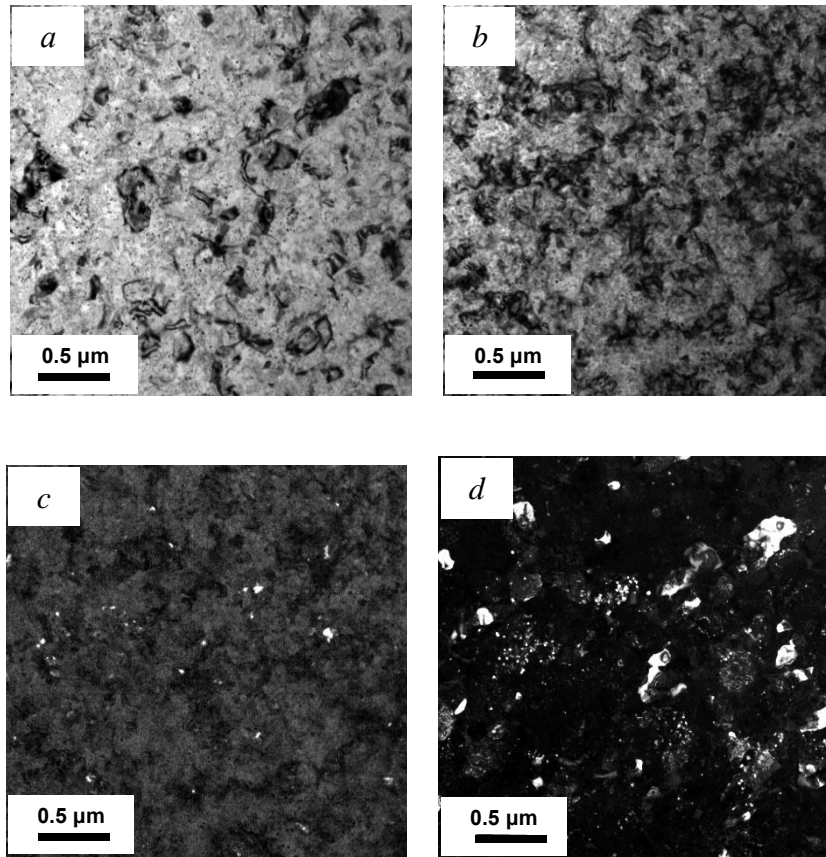


Figure 2. Microstructure of the 1469 alloy after SPD and Ar^+ ion irradiation, $F = 6.25 \cdot 10^{15} \text{ cm}^{-2}$: (*a*) (*b*) bright-field image and (*c*), (*d*) dark-field images: (*c*) in reflection $(530)_{\text{T}_2}$ and (*d*) in reflections $(111)_{\text{Al}}$ and $(530)_{\text{T}_2}$.

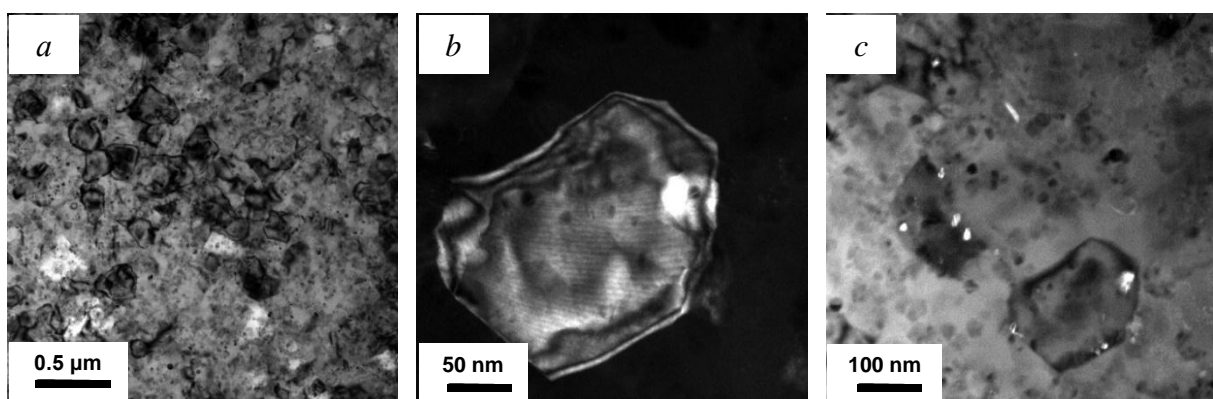


Figure 3. Microstructure of the 1469 alloy after SPD and Ar^+ ion irradiation, $F = 2 \cdot 10^{16} \text{ cm}^{-2}$: (*a*) bright-field image and (*b*), (*c*) dark-field images: (*b*) in reflection $(200)_{\text{Al}}$ and (*c*) in reflection $(530)_{\text{T}_2}$.

There are heterogeneously nucleated T_2 -phase particles at submicrocrystal boundaries. Similar to the first case, there is a bimodality of T_2 -phase precipitate size distribution. An increase in the fluence causes particle enlargement. The majority of the T_2 -phase particles have a diameter between 10–15 nm, whereas the diameter of some precipitates reaches 30 nm.

A comparison of microstructures in severely deformed 1469 alloy in states after short-term irradiation (during 10–32 s) and after low-temperature annealing (160 °C, 15 h) indicates a significant acceleration of structural-phase transformations during irradiation. A nanocrystalline structure is shown to remain in the 1469 alloy after prolonged annealing [6]. The size of nanograins formed during annealing-induced recrystallization, while some coarser grains tend to grow, is almost at the same level as after SPD. Annealing did not change the phase composition of the alloy noticeably.

4. Conclusions

This work showed that 10-keV Ar⁺ ion irradiation has an influence on the 1469 alloy structure subjected to SPD. The nature and degree of influence depend on the irradiation conditions. The increase in the ion fluence from $6.25 \cdot 10^{15} \text{ cm}^{-2}$ to $2 \cdot 10^{16} \text{ cm}^{-2}$ results in the transition from the mainly recrystallized nanocrystalline structure to the completely recrystallized submicrocrystalline structure. The irradiation liquidates the banded structure found in the alloy after SPD. Therefore, the structural elements (nanofragments, nanograins, submicrocrystals) are distributed uniformly in the volume of the irradiated sample. In addition, irradiation increases the volume fraction and the size bimodality of heterogeneously-generated T₂-phase particles. The mechanism of nucleation and growth of excess phases was proposed.

Structural and phase transformations during ion irradiation of the severely deformed alloy take place at a higher rate than those during traditional thermal annealing. They occur at a depth of ~200 µm from the irradiated surface, which significantly exceeds the projected range of argon ions in the alloy under study (~10 nm). These transformations can be explained on the basis of ideas about the radiation-dynamic effect of corpuscular radiation on metastable media [1, 2].

Acknowledgments

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